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## INFLUENCE OF MEASURING CONDITIONS ON THE QUANTIFICATION OF SPECTROSCOPIC SIGNALS IN TA-FTIR-MS SYSTEMS

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Simultaneous thermal analysis (TA) and evolved gas analysis by mass spectrometry (MS) and/or Fourier transform infrared spectroscopy (FTIR) is a powerful hyphenated technique combining direct measurement of mass loss and sensitive spectroscopic analysis. In the present study the influence of several experimental parameters which may affect the quantification of FTIR signals have been studied using a combined TA-FTIR-MS system. Parameters studied include: sample mass (1–400 mg), carrier gas flow rate (25–200 mL min<sup>-1</sup>), resolution of the FTIR spectrometer (1–32 cm<sup>-1</sup>), and location of injection of the calibrating gas.

MS analysis, which was not significantly affected by the experimental conditions, was used as a reference for assessing the accuracy of quantification by FTIR. The quantification of the spectroscopic signals was verified by the decomposition (NaHCO<sub>3</sub>) or dehydration (CuSO<sub>4</sub>·5H<sub>2</sub>O) of compounds with well-known stoichiometry.

The systematic study of the parametric sensitivity revealed that spectral resolution and carrier gas flow rate, which affect the acquisition time in the IR-cell, are key parameters that must be adjusted carefully for reliable quantification. The dependence of the reliability of quantification on these parameters is illustrated and conditions leading to proper quantification are discussed. As an example, for a standard spectral resolution of 4 cm<sup>-1</sup> and a FTIR gas cell volume of 8.7 mL, the carrier gas flow must be lower than 100 mL min<sup>-1</sup> for warranting accurate results (relative deviation <2%). The concentration range of analyzed species is limited but can be extended by proper selection of the wavenumber regions for molecules giving strong IR signals.

**Keywords:** calibration techniques, hyphenated techniques, pulse TA combined with FTIR, quantitative evolved gas analysis, TA-FTIR-MS

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